FORM-PTO-1390 (Rev. 9-2001) U.S DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371

ATTORNEY'S DOCKET NUMBER: 022650-690

U S APPLICATION NO. (If known, see 37 C F R 1 5): 10/070457

INTERNATIONAL APPLICATION NO.	INTERNATIONAL FILING DATE	PRIORITY DATE CLAIMED
PCT/FR00/02460	September 7, 2000	September 7, 1999

TITLE OF INVENTION: METHOD OF CONTROLLING SOIL INSECTS WITH PHENYLPYRAZOLES

APPLICANT(S) FOR DO/EO/US: Jean-Michel GAULLIARD and Christian SEGAUD

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This is an express request to begin national examination procedures (35 U.S.C. 371(f)). The submission must include items (5), (6), (9) and (21) indicated below.
4. ☒ The US has been elected by the expiration of 19 months from the priority date (Article 31).
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2)), including the Request and International Search Report.
   a. ☒ is attached hereto (required only if not communicated by the International Bureau).
   b. ☐ has been communicated by the International Bureau.
   c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☒ An English language translation of the International Application as filed (35 U.S.C. 371(c)(2)), including the Request and International Search Report.
   a. ☒ is attached hereto.
   b. ☐ has been previously submitted under 35 U.S.C. 154(d)(4).
7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
   a. ☐ are attached hereto (required only if not communicated by the International Bureau).
   b. ☐ have been communicated by the International Bureau.
   c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
   d. ☒ have not been made and will not be made.
8. ☐ An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☐ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. ☐ An English language translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11 to 20 below concern document(s) or information included:

11. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A FIRST preliminary amendment.
14. ☐ A SECOND or SUBSEQUENT preliminary amendment.
15. ☐ A substitute specification.
16. ☐ A change of power of attorney and/or address letter.
17. ☐ A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.821 - 1.825.
18. ☐ A second copy of the published international application under 35 U.S.C. 154(d)(4).
19. ☐ A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4).
20. ☒ Other items or information:

Separate Claim for Convention Priority and of Form PCT/IB/304 (in French).
Form PCT/IB/308.
International Preliminary Examination Report (Form PCT/IPEA/409) (in French and English).

The executed Declaration of the inventors will follow.

21839

JC13 Rec'd PCT/PTO 07 MAR 2002

U.S. APPLICATION NO. (If known, see 37 C.F.R. 1.5)	INTERNATIONAL APPLICATION NO	ATTORNEY'S DOCKET NUMBER
10/070457	PCT/FR00/02460	022650-690

21. ☒ The following fees are submitted:	CALCULATIONS	PTO USE ONLY
Basic National Fee (37 CFR 1.492(a)(1)-(5)):		
Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO $1,040.00 (960)		
International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO $890.00 (970)		
International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO $740.00 (958)		
International preliminary examination fee (37 CFR 1.482) paid to USPTO but all claims did not satisfy provisions of PCT Article 33(1)-(4) $710.00 (956)		
International preliminary examination fee (37 CFR 1.482) paid to USPTO and all claims satisfied provisions of PCT Article 33(1)-(4) $100.00 (962)		
ENTER APPROPRIATE BASIC FEE AMOUNT =	$ 890.00	
Surcharge of **$130.00 (154)** for furnishing the oath or declaration later than 20 ☐ 30 ☒ months from the earliest claimed priority date (37 CFR 1.492(e)).	$ 130.00	

Claims	Number Filed	Number Extra	Rate		
Total Claims	33 -20 =	13	X$18.00 (966)	$ 234.00	
Independent Claims	1 -3 =	0	X$84.00 (964)	$ 0.00	
Multiple dependent claim(s) (if applicable)			+$280.00 (968)	$ 0.00	
TOTAL OF ABOVE CALCULATIONS =				$ 1,254.00	
Reduction for 1/2 for filing by small entity, if applicable (see below).			+	$ 0.00	
SUBTOTAL =				$ 1,254.00	
Processing fee of **$130.00 (156)** for furnishing the English translation later than 20 ☐ 30 ☐ months from the earliest claimed priority date (37 CFR 1.492(f)).			+	$	
TOTAL NATIONAL FEE =				$ 1,254.00	
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). **$40.00 (581)** per property			+	$ 0.00	
TOTAL FEES ENCLOSED =				$ 1,254.00	
				Amount to be refunded:	$
				charged:	$

a. ☐ Small entity status is hereby claimed.

b. ☒ A check in the amount of $ 1,254.00 to cover the above fees is enclosed.

c. ☐ Please charge my Deposit Account No. 02-4800 in the amount of $_____ to cover the above fees. A duplicate copy of this sheet is enclosed.

d. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 02-4800. A duplicate copy of this sheet is enclosed.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:

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George F. Lesmes
SIGNATURE

George F. Lesmes
NAME

19,995
REGISTRATION NUMBER

March 7, 2002
DATE

Patent Application
Attorney's Docket No. 022650-690

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE
UNITED STATES ELECTED OFFICE (EO/US)
UNDER THE PATENT COOPERATION TREATY

In re Patent Application of	)	
	)	
Jean-Michel GAULLIARD et al	)	Group Art Unit:
	)	
International Application No.: PCT/FR00/02460	)	Examiner:
	)	
International Filing Date: September 7, 2000	)	
	)	
U.S. Application No.	)	
	)	
Date of Commencement of U.S. National Phase: March 7, 2002	)	
	)	
For: METHOD OF CONTROLLING SOIL INSECTS WITH PHENYLPYRAZOLES	)	

PRELIMINARY AMENDMENT

BOX PCT
Assistant Commissioner for Patents
Washington, D.C. 20231

Sir:

Prior to examination on the merits, please amend the accompanying application filed under 35 U.S.C. § 371 as follows:

IN THE SPECIFICATION:

Page 1, prior to line 3, add --BACKGROUND OF THE INVENTION--.

Page 1, after line 8, add --Description of Related Art--.

Page 2, after line 23, add --SUMMARY OF THE INVENTION--.

Page 5, after line 10, add --DESCRIPTION OF PREFERRED EMBODIMENTS--.

IN THE CLAIMS:

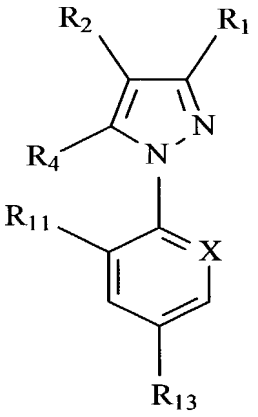
Page 17, at the top of the English translation of the published PCT application, amend the heading for the claims to read:

--WHAT IS CLAIMED IS:--

Please cancel Claims 1-17 and add the following new Claims 18-50:

--18. (New) Insecticidal compositions comprising:

(a) between 0.001 and 5 % of a compound of formula (I):



(I)

in which:

R_1 is a halogen atom or a CN group or a methyl group or a CH_3CO group;

R_2 is $S(O)_nR_3$;

R_3 is alkyl or haloalkyl;

R_4 represents a hydrogen or halogen atom, or an NR_5R_6, $S(O)_mR_7$, $C(O)R_7$ or $C(O)O\text{-}R_7$, alkyl, haloalkyl or OR_8 radical or an $-N=C(R_9)(R_{10})$ radical;

R_5 and R_6 independently represent a hydrogen atom or an alkyl, haloalkyl, C(O)alkyl or $S(O)_rCF_3$ radical, or R_5 and R_6 can together form a divalent alkylene radical optionally interrupted by one or two divalent heteroatoms;

R_7 represents an alkyl or haloalkyl radical;

R_8 represents an alkyl or haloalkyl radical or a hydrogen atom;

R_9 represents an alkyl radical or a hydrogen atom;

R_{10} represents a phenyl or heteroaryl group optionally substituted with at least one halogen atom or groups selected from OH, -O-alkyl, -S-alkyl, cyano or alkyl;

X represents a trivalent nitrogen atom or a $C\text{-}R_{12}$ radical, the other three valences of the carbon atom forming part of the aromatic ring;

R_{11} and R_{12} represent, independently of each other, a hydrogen or halogen atom;

R_{13} represents a halogen atom or a haloalkyl, haloalkoxy, $S(O)_qCF_3$ or SF_5 group;

m, n, q, r represent, independently of each other, an integer equal to 0, 1 or 2;

with the proviso that when R_1 is methyl, then R_3 is haloalkyl, R_4 is NH_2, R_{11} is Cl, R_{13} is CF_3 and X is N;

(b) between 0.05 and 10 % of at least one moisture-retaining agent; and

(c) between 40 and 99% of at least one vegetable meal.

19. (New) Insecticidal compositions according to Claim 18, wherein the vegetable meal is derived from the grinding of a cereal grain.

20. (New) Insecticidal compositions according to Claim 18, wherein the vegetable meal is a maize.

21. (New) Insecticidal compositions according to Claim 18, wherein the moisture-retaining agent is of an organic nature.

22. (New) Insecticidal compositions according to Claim 18, wherein the composition also comprises from 3 to 30 % of a sugar.

23. (New) Insecticidal compositions according to Claim 22, wherein the sugar is selected from mono, oligo- or polyorganosaccharides.

24. (New) Insecticidal compositions according to Claim 18, further comprising a preservative to prevent degradation of the meal.

25. (New) Insecticidal compositions according to Claim 18, further comprising one or more additives selected from colorings, attractants for pests, repellants for birds or animals which are useful or which should be protected, binding agents, agglomerating

agents, appetite-enhancing agents, agglutinating agents, gelling agents, swelling agents or antiadherent agents.

26. (New) Insecticidal compositions according to Claim 18, wherein the compound of formula (I) is 5-amino-3-cyano-1-[2,6-di-chloro-4-(trifluoromethyl)phenyl]-4-[(trifluoromethyl)-sulphinyl]-1H-pyrazole.

27. (New) Insecticidal compositions according to Claim 18, which are in the form of granules of a size between 0.1 mm and 3 cm.

28. (New) Insecticidal compositions according to Claim 18, wherein the compound of formula (I) is present in an amount of between 0.05 and 1 %.

29. (New) Insecticidal compositions according to Claim 28, wherein the compound of formula (I) is present in an amount of between 0.05 and 0.5 %.

30. (New) Insecticidal compositions according to Claim 18, wherein the moisture-retaining agent is present in an amount of between 0.1 and 5 %.

31. (New) Insecticidal compositions according to Claim 18, wherein the vegetable meal is present in an amount of between 50 and 98 %.

32. (New) Insecticidal compositions according to Claim 31, wherein the vegetable meal is present in an amount of between 70 and 97 %.

33. (New) Insecticidal compositions according to Claim 19, wherein said cereal grain is selected from wheat, barley, rye, triticale, oats, rice, sorghum, soyabean or maize.

34. (New) Insecticidal compositions according to Claim 21, wherein the moisture-retaining agent of an organic nature is selected from macromolecular hydrophilic derivatives of plant origin.

35. (New) Insecticidal compositions according to Claim 34, wherein the moisture-retaining agent is a cellulosic hydrophilic derivative.

36. (New) Insecticidal compositions according to Claim 22 wherein the sugar is present in an amount of between 4 to 20 %.

37. (New) Insecticidal compositions according to Claim 23, wherein the sugar is sucrose, lactose, fructose, dextrose, glucose, molasses or honey.

38. (New) Insecticidal compositions according to Claim 24, wherein the preservative is selected from sodium benzoate, 1,2-benziso-thiazolin-3-one, benzoic acid,

para-hydroxybenzoic acid and its esters and alkali or alkaline-earth metal salts, 2-phenylphenol and its alkali or alkaline-earth metal salts, or para-nitrophenol.

39. (New) Insecticidal compositions according to Claim 27, wherein the granules are of a size between 0.5 and 4 mm and are water-insoluble.

40. (New) Insecticidal compositions according to Claim 18, further including from 0.5 to 30 % of at least one disintegrating agent.

41. (New) Insecticidal compositions according to Claim 40, wherein the disintegrating agent is present in an amount of 1 to 20 % and is selected from starch, sodium carboxymethyl starch, microcrystalline cellulose; modified celluloses; bentonite, aluminium or magnesium silicate; sodium polynaphthalenesulphonate, sodium dodecylbenzenesulphonate, sodium dioctylsulphosuccinate, lignin sulphonate; a saccharide derivative; or a cross-linked derivative of polyvinylpyrrolidone.

42. (New) A method of controlling insects which comprises applying an effective quantity of a composition according to Claim 18 in the form of granules having a size of between 0.2 mm and 2 cm over or into the soil in an area which has to be cultivated.

43. (New) A method according to Claim 42, wherein the effective quantity is selected to provide a dosage which is nonlethal through contact but lethal through ingestion.

44. (New) A method of protecting crops which comprises applying an effective amount of a composition according to Claim 18 in the form of granules.

45. (New) A method according to Claim 44, wherein the crop to be protected is selected from a cereal, beet, sunflower, potato or rape.

46. (New) A method according to Claim 42, wherein the insect being controlled is a click beetle.

47. (New) A method according to Claim 44, wherein the effective amount comprises between 1 and 50 g/ha.

48. (New) A method according to Claim 47, wherein the amount is between 3 and 40 g/ha.

49. (New) A method according to Claim 42, wherein the effective quantity is between 1 and 50 g/ha.

50. (New) A method according to Claim 49, wherein the effective quantity is between 3 and 40 g/ha.

IN THE ABSTRACT OF THE DISCLOSURE:

Please add the abstract presented on the accompanying separate page 22 to replace the abstract appearing on the cover of the English translation of the published version of the PCT application.

REMARKS

Entry of the foregoing and prompt and favorable consideration of the subject application, in light of the following remarks, are respectfully requested.

The foregoing amendments have been made to place the claims in better form for examination and eliminate multiple dependent claim terminology. Claims 1-17 have been canceled without prejudice or disclaimer to the subject matter recited therein and new Claims 18-50 have been added. Support for Claims 18-50 can be found in canceled Claims 1-17 and throughout the specification of the subject application. No new matter has been added by the foregoing amendment.

In the event that there are any questions relating to this Preliminary Amendment, or the application in general, it would be appreciated if the Examiner would telephone the undersigned concerning such questions so that prosecution of this application may be expedited.

Respectfully submitted,

BURNS, DOANE, SWECKER & MATHIS, L.L.P.

By: _____
George F. Lesmes
Registration No. 19,995

Post Office Box 1404
Alexandria, Virginia 22313-1404
(703) 836-6620

Date: March 7, 2002

Attachment to Preliminary Amendment dated March 7, 2002

Marked-up Claims

Claims heading on page 17:

[CLAIMS]WHAT IS CLAIMED IS:

ABSTRACT OF THE DISCLOSURE

The invention concerns insecticide compositions comprising:

(a) between 0.001 and 5 %, preferably between 0.05 and 1 % of a compound of the type 1-phenyl pyrazole, in particular 1-[2,6-$Cl_2$4-CF_3 phenyl]3-CN 4-[SO-CF_3]5-NH_2 pyrazole; and

(b) between 0.05 and 10 %, preferably between 0.1 and 5 % of one (or several) moisture retaining agent of the organic type; and

(c) between 40 and 99 %, preferably between 50 and 98 % (and more preferably between 70 and 97 %) of vegetable flour.

The invention also concerns a method for fighting insects using said compositions, in particular against click beetles.

Method of controlling soil insects with phenylpyrazoles

The subject of the present invention is novel compositions intended for controlling soil insects in their various developmental forms, and in particular compositions useful for controlling click beetles. The invention also relates to a method of control using the said compositions.

Insecticidal compounds of the phenylpyrazole type which can be used in controlling insects are known in particular from patent applications EP 295117, WO 87/3781, 93/6089 and 94/21606. Patent applications EP 295117 and 836386 also mention compositions comprising from 0.01% to 5% of such active substances.

Click beetles constitute a family of insects which are particularly harmful for certain crops, more particularly for maize, beet, sunflower, potato and rape crops. Their harmful character is all the more marked since the larval forms of click beetles can remain for very long periods in the soil, extending up to 5 years.

Baits have indeed been proposed for various sorts of insects, as well as formulas which can be consumed by ingestion, but these formulas are not necessarily active for all the types of insect and the need remains to find insecticidal forms or formulations which are particularly effective for the most diverse

applications, and in particular for controlling click beetles.

In addition, as regards the insecticides applied over or into the soil, it is desirable to find conditions and formulations which make it possible to obtain good efficacy at doses which are as low as possible.

One aim of the invention is to overcome these difficulties completely or in part.

Another aim of the invention is to provide advantageous and effective compositions for controlling non-gregarious insects.

Another aim of the invention is to provide advantageous and effective compositions for controlling soil insects, especially click beetles, and more particularly click beetles in the larval state.

Another aim of the invention is to provide compositions comprising at least one insecticidal active substance of the phenylpyrazole type and which are easily applicable over or into the soil.

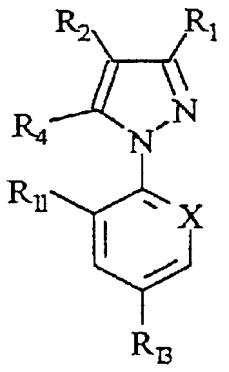
Another aim of the invention is to provide insecticide compositions whose performance is good in spite of low applicable doses.

It has now been found that these aims could be achieved, completely or in part, by means of the compositions and the control method according to the invention which are described in detail below. It is

specified that the percentages indicated in the present text are weight/weight percentages, unless otherwise indicated.

The subject of the present invention is therefore, firstly, insecticidal compositions comprising:

- a) between 0.001 and 5%, preferably between 0.05 and 1% and still more advantageously between 0.05 and 0.5% of the compound of formula (I):



(I)

in which:

R_1 is a halogen atom or a CN group or a methyl group or a CH_3CO group;

R_2 is $S(O)_nR_3$;

R_3 is alkyl or haloalkyl;

R_4 represents a hydrogen or halogen atom, or an NR_5R_6, $S(O)_mR_7$, $C(O)R_7$ or $C(O)O-R_7$, alkyl, haloalkyl or OR_8 radical or an $-N=C(R_9)(R_{10})$ radical;

R_5 and R_6 independently represent a hydrogen atom or an alkyl, haloalkyl, C(O)alkyl or $S(O)_rCF_3$

radical, or R_5 and R_6 can together form a divalent alkylene radical which may be interrupted by one or two divalent heteroatoms such as oxygen or sulphur;

R_7 represents an alkyl or haloalkyl radical;

R_8 represents an alkyl or haloalkyl radical or a hydrogen atom;

R_9 represents an alkyl radical or a hydrogen atom;

R_{10} represents a phenyl or heteroaryl group optionally substituted with one or more halogen atoms or groups such as OH, -O-alkyl, -S-alkyl, cyano or alkyl;

X represents a trivalent nitrogen atom or a C-R_{12} radical, the other three valencies of the carbon atom forming part of the aromatic ring;

R_{11} and R_{12} represent, independently of each other, a hydrogen or halogen atom;

R_{13} represents a halogen atom or a haloalkyl, haloalkoxy, $S(O)_qCF_3$ or SF_5 group;

m, n, q, r represent, independently of each other, an integer equal to 0, 1 or 2;

with the proviso that when R_1 is methyl, then R_3 is haloalkyl, R_4 is NH_2, R_{11} is Cl, R_{13} is CF_3 and X is N;

- b) between 0.05 and 10%, preferably between 0.1 and 5% of one (or more) moisture-retaining agents, preferably a moisture-retaining agent of an organic

nature; and

- c) between 40 and 99%, preferably between 50 and 98% (and more preferably between 70 and 97%) of vegetable meal.

The alkyl radicals of the definition of formula (I) generally comprise from 1 to 6 carbon atoms. The ring formed by the divalent alkylene radical representing R_5 and R_6 as well as by the nitrogen atom to which R_5 and R_6 are attached is generally a 5-, 6- or 7-membered ring.

The compound of formula (I) may be prepared according to one of the methods described in patent applications WO 87/3781, 93/6089, 94/21606, EP 295117 or alternatively by another method within the general knowledge of persons skilled in the art competent in chemical synthesis. This compound is generally designated in the present text by the term active substance.

Among the vegetable meals which can be used, there may be mentioned the meals derived from the grinding of cereal grains such as wheat, barley, rye, triticale, oats, or also rice, sorghum, soyabean, maize, the preferred meal being that based on maize. A mixture of these vegetable meals can also be envisaged in the context of the present invention.

Among the moisture-retaining agents of an organic nature, there may be mentioned the

macromolecular hydrophilic derivatives of plant origin, and in particular the cellulosic hydrophilic derivatives, and more particularly cellulose, but also one or more disintegrating agents. It may be advantageous to use these compounds in particular when meals such as hard wheat meals are used in the granules. Disintegrating agents include: starch, sodium carboxymethyl starch, cellulose such as microcrystalline cellulose; modified celluloses such as sodium carboxymethylcellulose; bentonite, aluminium and magnesium silicate; sodium polynaphthalenesulphonate, sodium dodecylbenzenesulphonate, sodium dioctylsulphosuccinate, lignin sulphonate; a saccharide derivative such as lactose, fructose, sucrose, mannitol, dextrose; a cross-linked derivative of polyvinylpyrrolidone. When a disintegrating agent is used, the composition according to the invention, may contain from 0.5 to 30%, and preferably from 1 to 20%, by weight of the dry substance, of the said agent(s).

According to a variant of the composition according to the invention, the composition also comprises from 3 to 30%, preferably from 4 to 20% of sugars. The sugars are chosen in particular from mono-, oligo- or polyorganosaccharides, especially from sucrose, lactose, fructose, dextrose, glucose or alternatively molasses or honey.

The compositions which are the subject of the

invention may also comprise a preservative preventing the degradation of the meals, such as sodium benzoate, 1,2-benzisothiazolin-3-one, benzoic acid, para-hydroxybenzoic acid and its ester derivatives and its alkali or alkaline-earth metal salts, in particular the sodium salt, 2-phenylphenol and its alkali or alkaline-earth metal salts, in particular the sodium salt, para-nitrophenol.

Other additives may also be included such as colourings or attractants for pests or repellents for birds or animals which are useful or which should be protected.

Other formulation additives may be used such as binding, agglomerating, appetite-enhancing, agglutinating, gelling, swelling or antiadherent agents and the like.

A preferred class of compounds of formula (I) comprises compounds such that R_1 is CN, and/or R_3 is haloalkyl, and/or R_4 is NH_2, and/or R_{11} and R_{12} are, independently of each other, a halogen atom, and/or R_{13} is haloalkyl.

According to a particularly advantageous variant of the invention, the compound of formula (I) used in the invention is 5-amino-3-cyano-1-[2,6-dichloro-4-(trifluoromethyl)phenyl]-4-[(trifluoromethyl)-sulphinyl]-1*H*-pyrazole, to which reference is made in the examples under the term "fipronil".

The formulations according to the invention are generally in the form of granules. The size of the granules is advantageously between 0.1 mm and 3 cm, preferably between 0.5 and 4 mm. These granules are advantageously insoluble in water (in the sense that they resist disintegration with water).

The compositions according to the invention may be prepared by simply mixing the various constituents, preferably by extrusion or compression in the cold or hot state according to any granulation or pelleting technique known per se. For the production of such granules, reference can be easily made to the European patent application published under the number EP 0575838 and/or to other techniques, for example extrusion techniques, known to persons skilled in the art.

The invention also relates to a method of protecting crops from insects, especially click beetles, characterized in that an effective quantity of a composition in the form of granules having a size of between 0.2 mm and 2 cm comprising an active substance chosen from the group consisting of the products of formula I, imidacloprid, acetamiprid, nitenpyram and thiamethoxam, is applied over or into the soil (preferably into the soil) of the area which has to be cultivated.

The invention thus relates more particularly

to a method of protecting cereal, preferably maize or beet or sunflower or potato or rape, crops. The application of the formulations according to the invention takes place advantageously before sowing the said crop, or simultaneously with this sowing.

The invention also relates to a method of controlling insects, especially click beetles, characterized in that an effective quantity of one of the compositions according to the invention is applied over or into the soil (preferably into the soil) where they are present or are likely to be present.

As effective quantity, quantities of composition corresponding to a dose of compound of formula (I) of between 1 and 50 g/ha, preferably between 3 and 40 g/ha are often used.

A specific characteristic of the method of controlling insects according to the invention consists in the application, over or into the soil, of a composition providing a dose which is nonlethal through contact but lethal through ingestion.

In other words, in the specific case of click beetles, the method consists in killing the click beetles by application of a dose which is nonlethal through contact but lethal through ingestion. A hypothesis for the good efficacy of the method of treatment according to the invention, which makes it possible to greatly reduce the applicable doses of

compounds of formula I in particular, is based on the fact that once the bait according to the invention has caused the death of a click beetle, the latter can itself serve as bait for other click beetles, which therefore also ingest a product (dead click beetle) containing the insecticide.

For the purposes of the present text, the words insecticide and insect should be taken in their broad ordinary sense and not in their strictly scientific (zoological) sense. Accordingly, the term insect is understood to mean any animal of a very small size such as arthropods (insects in the strict and zoological sense, arachnids, myriapods) and nematodes.

As soil insects against which the invention is particularly effective, there may be mentioned for example:

The **Coleoptera** (wireworms (*Agriotes* spp.), false wireworms, white grubs) such as for example:

Agriotes lineatus (European click beetle, Elateridae),

Agriotes sordidus (European click beetle, Elateridae),

Agriotes obscurus (European click beetle, Elateridae),

Agriotes sputator (European click beetle, Elateridae),

Athous spp. (Elateridae),

Atomaria linearis (Cryptophagidae)

Melolontha spp. (white grubs, Scarabaeidae),

Bothynoderes

Limonius spp. (US click beetle),

Melanotus spp. (US click beetle),

Diabrotica spp. (cornrootworms, Crysomelidae),

Tanymecus pallidus (beet leaf weevil, Curculionidae).

The **Lepidoptera** (Noctuidae) such as:

Autographa spp., *Mamestra* spp., *Agrotis* spp. (cutworms, grey grubs), *Euxoa* spp. (cutworms, grey grubs), *Spodoptera* spp. (*Spodoptera exigua, Spodoptera littorlis*).

The **Diptera** such as *Tipula* spp.).

The **Myriapoda** (Myriapoda):

- *Diplopoda* = Millipedes,
- Centipede.

Among the soil click beetles against which the invention is particularly effective, there may be mentioned *Agriotes* spp., *Athous* spp., *Limonius* spp.

The granules according to the invention are advantageously inserted into the soil at a depth of between 1 and 5 cm.

The compositions according to the invention are particularly advantageous in that they allow the use of lower doses of active product than similar known compositions.

The following examples illustrate the invention without however constituting a limitation thereto. In these examples, the compound of formula (I) used is fipronil.

Example 1:

A surface of 0.1 ha is sown with maize at the rate of about 8000 untreated seeds. This surface is divided into 40-m^2 plots.

At the same time as the sowing, there are incorporated into the soil, in the sowing row, 2 mm granules containing a composition consisting of:

0.25% of fipronil,

93.5% of maize meal,

2% of cellulose,

4% of lactose,

0.2% of para-nitrophenol,

0.05% of pigment blue 15.3.

The quantities of granules thus spread vary from 2.5 to 10 kg per hectare. Untreated plots are kept to serve as control and to verify the extent of the damage by the insects. Likewise, the plots will be treated with a commercial insecticide which is reputed effective and called reference. Each modality is repeated four times.

Approximately 20 days after sowing, the maize plants which have emerged are counted.

In the locality of St Hilaire (30), 22 days

after sowing, 19 plants are observed in the furrow of the 4 untreated plots per 10 metres of furrow. In the plots treated according to the invention with the dose of fipronil of 6 g per hectare, 49 plants are observed per 10 metres of furrow.

With the dose of fipronil of 12.5 g per hectare, 48 to 50.5 plants are observed per 10 metres of furrow.

With the dose of fipronil of 25 g per hectare, 45.5 to 51.3 plants are observed per 10 metres of furrow.

With the dose of fipronil of 50 g per hectare, 49 plants are observed per 10 metres of furrow.

To obtain the same result with a conventional granule (clay carrier), 200 g of fipronil have to be provided per hectare.

The soil is dug out and scraped in order to capture and identify the insects responsible for the damage; a large presence of larvae of click beetles of the genus Agriotes, in particular Agriotes sordidus, is observed for the untreated control.

Example 2:

A surface of 1 ha is sown with maize at the rate of about 98,100 untreated seeds. This surface is divided into 27-m^2 plots.

At the same time as the sowing, there are

incorporated into the soil, in the sowing row, 2 mm granules containing a composition consisting of:

0.25% of fipronil,

93.5% of maize meal

2% of cellulose,

4% of lactose,

0.2% of para-nitrophenol,

0.05% of pigment blue 15.3.

The quantities of granules thus spread vary from 2.5 to 10 kg per hectare. Untreated plots are kept to serve as control and to verify the extent of the damage by the insects. Likewise, the plots will be treated with a commercial insecticide which is reputed effective and called reference. Each modality is repeated four times.

Approximately 50 days after sowing, the maize plants which have emerged are counted.

In the locality of Beaufort (62), 50 days after sowing, 30 plants are observed in the furrow of the 4 untreated plots per 10 metres of furrow. In the plots treated according to the invention with the dose of fipronil of 6 g per hectare, 40 plants are observed per 10 metres of furrow.

With the dose of fipronil of 12.5 g per hectare, 43 to 46 plants are observed per 10 metres of furrow.

With the dose of fipronil of 25 g per

hectare, 44.5 to 50 plants are observed per 10 metres of furrow.

With the dose of fipronil of 50 g per hectare, 49.8 plants are observed per 10 metres of furrow.

To obtain the same result (52 plants per 10 metres) with a conventional granule (clay carrier), 200 g of fipronil have to be provided per hectare.

The soil is dug out and scraped in order to capture and identify the insects responsible for the damage; a large presence of larvae of click beetles of the genus Agriotes, in particular Agriotes lineatus.

Example 3: Test of efficacy of the bait granules according to the invention on potato crop.

At the same time as the sowing, there are incorporated into the soil, in the sowing row, granules according to the invention consisting of:

12.5 g/kg of fipronil (from an SC containing 500 g/l of fipronil),

945.5 g/kg of a) **maize** meal (**granules A**) or b) **rice** meal (**granules B**),

40 g/kg of lactose,

2 g/kg of para-nitrophenol.

The efficacy against Agriotes (click beetles), 90 days after sowing, of the two granules A and B above, used at the doses of 5 and 2.5 g of fipronil per hectare, and of the same fipronil used by

spraying at the doses of 50 and 25 g/ha (from Regent® 800WG) (compound C), was compared.

The following results, expressed as number of holes per 10 tubers (= N) are then observed:

Product	A	A	B	B	C	C	Control*
Dose (g.ha)	2.5	5	2.5	5	25	50	-
N	6	1	7	3	6	1	14

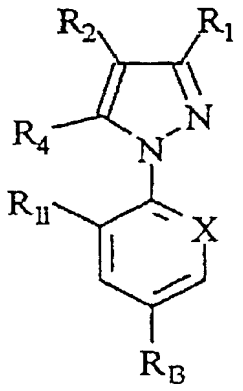
* untreated control.

This result indeed shows the good control of click beetles which is obtained by the granules according to the invention which give the same result as Regent® 800WG **but with a dose reduced by a factor of 10.**

CLAIMS

1. Insecticidal compositions comprising:

- a) between 0.001 and 5%, preferably between 0.05 and 1% and still more advantageously between 0.05 and 0.5% of a compound of formula (I):



(I)

in which:

R_1 is a halogen atom or a CN group or a methyl group or a CH_3CO group;

R_2 is $S(O)_nR_3$;

R_3 is alkyl or haloalkyl;

R_4 represents a hydrogen or halogen atom, or an NR_5R_6, $S(O)_mR_7$, $C(O)R_7$ or $C(O)O-R_7$, alkyl, haloalkyl or OR_8 radical or an $-N=C(R_9)(R_{10})$ radical;

R_5 and R_6 independently represent a hydrogen atom or an alkyl, haloalkyl, C(O)alkyl or $S(O)_rCF_3$ radical, or R_5 and R_6 can together form a divalent alkylene radical which may be interrupted by one or two

divalent heteroatoms such as oxygen or sulphur;

R_7 represents an alkyl or haloalkyl radical;

R_8 represents an alkyl or haloalkyl radical or a hydrogen atom;

R_9 represents an alkyl radical or a hydrogen atom;

R_{10} represents a phenyl or heteroaryl group optionally substituted with one or more halogen atoms or groups such as OH, -O-alkyl, -S-alkyl, cyano or alkyl;

R_{11} and R_{12} represent, independently of each other, a hydrogen or halogen atom;

R_{13} represents a halogen atom or a haloalkyl, haloalkoxy, $S(O)_qCF_3$ or SF_5 group;

m, n, q, r represent, independently of each other, an integer equal to 0, 1 or 2;

with the proviso that when R_1 is methyl, then R_3 is haloalkyl, R_4 is NH_2, R_{11} is Cl, R_{13} is CF_3 and X is N;

- b) between 0.05 and 10%, preferably between 0.1 and 5% of one (or more) moisture-retaining agents, preferably a moisture-retaining agent of an organic nature; and

- c) between 40 and 99%, preferably between 50 and 98% (and more preferably between 70 and 97%) of vegetable meal.

2. Insecticidal compositions according to

Claim 1, characterized in that the meals are derived from the grinding of cereal grains such as wheat, barley, rye, triticale, oats, or also rice, sorghum, soyabean, maize.

3. Insecticidal compositions according to either of Claims 1 and 2, characterized in that the meal is a maize meal.

4. Insecticidal compositions according to one of Claims 1 to 3, characterized in that among the moisture-retaining agents of an organic nature, there may be mentioned macromolecular hydrophilic derivatives of plant origin, and in particular cellulosic hydrophilic derivatives, and more particularly cellulose.

5. Insecticidal compositions according to one of Claims 1 to 4, characterized in that the composition also comprises from 3 to 30%, preferably from 4 to 20% of sugars.

6. Insecticidal compositions according to one of Claims 1 to 4, characterized in that the sugars are chosen in particular from mono-, oligo- or polyorganosaccharides, especially from sucrose, lactose, fructose, dextrose, glucose or alternatively molasses or honey.

7. Insecticidal compositions according to one of Claims 1 to 6, characterized in that they also comprise a preservative preventing the degradation of

the meals, chosen from sodium benzoate, 1,2-benziso-thiazolin-3-one, benzoic acid, para-hydroxybenzoic acid and its ester derivatives and its alkali or alkaline-earth metal salts, in particular the sodium salt, 2-phenylphenol and its alkali or alkaline-earth metal salts, in particular the sodium salt, para-nitrophenol.

8. Insecticidal compositions according to one of Claims 1 to 7, characterized in that they comprise other additives such as colourings or attractants for pests or repellants for birds or animals which are useful or which should be protected, and/or other formulation additives such as binding, agglomerating, appetite-enhancing, agglutinating, gelling, swelling and antiadherent agents and the like.

9. Insecticidal compositions according to one of Claims 1 to 8, characterized in that the compound of formula (I) is 5-amino-3-cyano-1-[2,6-di-chloro-4-(trifluoromethyl)phenyl]-4-[(trifluoromethyl)-sulphinyl]-1*H*-pyrazole.

10. Insecticidal compositions according to one of Claims 1 to 9, characterized in that the formulations according to the invention are in the form of granules, the size of these granules being advantageously between 0.1 mm and 3 cm, preferably between 0.5 and 4 mm and in that these granules are advantageously insoluble in water.

11. Method of controlling insects,

characterized in that an effective quantity of a composition according to one of Claims 1 to 10 in the form of granules having a size of between 0.2 mm and 2 cm is applied over or into the soil (preferably into the soil) of the area which has to be cultivated.

12. Method of protecting crops according to Claim 11, characterized in that a compound of formula I is used.

13. Method according to either of Claims 11 and 12, characterized in that cereal, preferably maize or beet or sunflower or potato or rape, crops are protected.

14. Method according to one of Claims 11 to 13, characterized in that an effective quantity of one of the compositions according to the invention is used for controlling insects, especially click beetles.

15. Method according to one of Claims 12 to 14, characterized in that the effective quantity of composition corresponds to a dose of compound of formula (I) of between 1 and 50 g/ha, preferably between 3 and 40 g/ha.

16. Method of controlling insects which consists in the application, over or into the soil, of a composition providing a dose which is nonlethal through contact but lethal through ingestion.

17. Method according to Claim 16, applicable to click beetles.

(12) DEMANDE INTERNATIONALE PUBLIÉE EN VERTU DU TRAITÉ DE COOPÉRATION EN MATIÈRE DE BREVETS (PCT)

(19) Organisation Mondiale de la Propriété Intellectuelle
Bureau international

(43) Date de la publication internationale
15 mars 2001 (15.03.2001)

PCT

(10) Numéro de publication internationale
WO 01/17354 A1

(51) Classification internationale des brevets[7]: A01N 47/02, 43/56 // (A01N 47/02, 25:12, 25:00) (A01N 43/56, 25:12, 25:00)

(21) Numéro de la demande internationale:
PCT/FR00/02460

(22) Date de dépôt international:
7 septembre 2000 (07.09.2000)

(25) Langue de dépôt: français

(26) Langue de publication: français

(30) Données relatives à la priorité:
99/11312 7 septembre 1999 (07.09.1999) FR

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(84) États désignés *(régional)*: brevet ARIPO (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), brevet eurasien (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), brevet européen (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), brevet OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

Publiée:
— *Avec rapport de recherche internationale.*

En ce qui concerne les codes à deux lettres et autres abréviations, se référer aux "Notes explicatives relatives aux codes et abréviations" figurant au début de chaque numéro ordinaire de la Gazette du PCT.

(54) **Title:** METHOD FOR FIGHTING SOIL INSECTS WITH PHENYL-PYRAZOLES

(54) **Titre:** PROCEDE DE LUTTE CONTRE LES INSECTES DU SOL AVEC DES PHENYL-PYRAZOLES

(57) **Abstract:** The invention concerns insecticide compositions comprising: a) between 0.001 and 5 %, preferably between 0.05 and 1 % of a compound of the type 1-phenyl pyrazole, in particular 1-[2,6-$Cl_2$4-CF_3 phenyl] 3-CN 4-[SO-CF_3] 5-NH_2 pyrazole; and b) between 0.05 and 10 %, preferably between 0.1 and 5 % of one (or several) moisture retaining agent of the organic type; and c) between 40 and 99 %, preferably between 50 and 98 % (and more preferably between 70 and 97%) of vegetable flour. The invention also concerns a method for fighting insects using said composition, in particular against click beetles.

(57) **Abrégé:** Compositions insecticides comprenant: a) entre 0,001 et 5 %, de préférence entre 0,05 et 1 % d'un composé de type 1-phényl pyrazole, notamment le 1-[2,6-$Cl_2$4-CF_3phenyl]3-CN4-[SO-CF_3]5-NH_2 pyrazole, et; b) entre 0,05 et 10 %, de préférence entre 0,1 et 5 % d'un (ou plusieurs) agent rétenteur d'humidité, de préférence un agent rétenteur d'humidité de nature organique, et; c) entre 40 et 99 %, de préférence entre 50 et 98 % (et plus préférentiellement entre 70 et 97 %) de farine végétale. Procédé de lutte contre les insectes mettant en oeuvre cette composition, notamment contre les taupins.

WO 01/17354 A1

10/USPTS PRO9905

022650-690
Attorney's Docket No.

COMBINED DECLARATION AND POWER OF ATTORNEY FOR UTILITY OR DESIGN PATENT APPLICATION

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name;

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

METHOD OF CONTROLLING SOIL INSECTS WITH PHENYLPYRAZOLES

the specification of which (check only one item below):

☐ is attached hereto.

☐ was filed as United States application
Number _____ on _____
and was amended on _____(if applicable).

☒ was filed as PCT international application
Number PCT/FR00/02460 on September 7, 2000
and was amended on _____(if applicable).

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose to the Office all information known to me to be material to patentability as defined in Title 37, Code of Federal Regulations, §1.56.

I hereby claim foreign priority benefits under Title 35, United States Code, §§119 (a)-(d), 172 or 365 of any foreign application(s) for patent or inventor's certificate or of any PCT international application(s) designating at least one country other than the United States of America listed below and have also identified below any foreign application(s) for patent or inventor's certificate or any PCT international application(s) designating at least one country other than the United States of America filed by me on the same subject matter having a filing date before that of the application(s) of which priority is claimed:

PRIOR FOREIGN/PCT APPLICATION(S) AND ANY PRIORITY CLAIMS UNDER 35 U.S.C. §§119(a)-(d), 172 or 365:				
COUNTRY (if PCT, indicate "PCT")	APPLICATION NUMBER	DATE OF FILING (day, month, year)	PRIORITY CLAIMED UNDER 35 U.S.C. §§119, 172 or 365	
FRANCE	99/11312	07 September 1999	X Yes	No
			Yes	No
			Yes	No
			Yes	No
			Yes	No

022650-690
Attorney's Docket No.

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FRANCE	99/11312	07 September 1999	X	Yes	No
				Yes	No
				Yes	No
				Yes	No
				Yes	No

Combined Declaration and Power of Attorney
for Utility or Design Patent Application
Attorney's Docket No. 022650-690

I hereby appoint the following attorneys and agent(s) to prosecute said application and to transact all business in the Patent and Trademark Office connected therewith and to file, prosecute and to transact all business in connection with international applications directed to said invention:

William L. Mathis	17,337	Eric H. Weisblatt	30,505	Bruce T. Wieder	33,815
Robert S. Swecker	19,885	James W. Peterson	26,057	Todd R. Walters	34,040
Platon N. Mandros	22,124	Teresa Stanek Rea	30,427	Ronni S. Jillions	31,979
Benton S. Duffett, Jr.	22,030	Robert E. Krebs	25,885	Harold R. Brown III	36,341
Norman H. Stepno	22,716	William C. Rowland	30,888	Allen R. Baum	36,086
Ronald L. Grudziecki	24,970	T. Gene Dillahunty	25,423	Brian P. O'Shaughnessy	32,747
Frederick G. Michaud, Jr.	26,003	Patrick C. Keane	32,858	Kenneth B. Leffler	36,075
Alan E. Kopecki	25,813	B. Jefferson Boggs, Jr.	32,344	Fred W. Hathaway	32,236
Regis E. Slutter	26,999	William H. Benz	25,952	Wendi L. Weinstein	34,456
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E. Joseph Gess	28,510	Gerald F. Swiss	30,113		
R. Danny Huntington	27,903	Charles F. Wieland III	33,096		

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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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FULL NAME SECOND INVENTOR, IF ANY	
Signature	Christian SEGAUD
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FULL NAME OF SOLE OR FIRST INVENTOR	Jean-Michel GAULLIARD
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